# MONITOR CIGARETTE:

# Making, Use, and Statistical Control

## CONTENTS

# MONITOR CIGARETTE

## Part I: Production of a Monitor Cigarette

The following describes the stages of preparation for the productions of monitor cigarettes:

1. Site Selection for Filler/Cigarette Production
2. Product Specifications/QC checks
3. Environmental Equilibration
4. Canning/Shipping
5. Equipment

### A. Site Selection: Filler/Cigarette Production

To ensure that the blend was highly uniform, a basic component formula was chosen that was double blended, checked throughout the process for oven volatiles (OV) and oversprayed with a generic flavor system. The filler may be pneumatically transferred to the cigarette maker or loaded into an appropriate container and manually fed to the machine. Care must be taken if the latter option is chosen to guard against possible filler degradation and breakage during the extra handling.

### B. Product Specifications/Quality Control

The product was chosen to have zero ventilation to minimize tar variation. Each individual bobbin of cigarette paper was prescreened in one of our laboratories prior to cigarette production. The accepted tolerances were tighter than normal vendor requirements. Tobacco weight was increased above conventional tobacco weight to ensure that the product was tightly packed, had a uniform density, and an acceptable firmness. The proper equipment mix was chosen (Mark 9/Max Tipper) and the Accu Ray weight control units were set to accept a target cigarette weight ±20 mg. During the production run, a random sample was selected from each pallet and tested for firmness, weight and weight histogram, as is OV and RTD. Production was being monitored by production technicians, supervisors, R&D professionals and Q/C engineers.

C. Equilibration

Production monitor cigarettes were sent to a specially controlled environmental room where strict adherence to FTC (75°F ± 1°F, 60% RH ± 2%) conditions were maintained. Samples were set on shelving which allowed good air flow for uniform equilibration. Laboratory conditions were monitored using Thunder Scientific wet bulb/dry bulb psychrometers and Honeywell digital temperature/RH recorders.

A series of random samples were drawn to test for OV throughout the equilibration period. As soon as stable conditions and uniform oven volatiles were achieved (approximately 2 weeks), the monitor was ready to be placed in cans.

D. Canning/Shipping

The equilibrated monitor is placed in cans (approximately 80/can). A Kimwipe is put into each can to ensure that the product remains intact during handling and shipping. After the can sealer crimps a lid on each can, the air-tight container is put into cold storage (2 weeks @ 34°F) to kill any tobacco beetles or infestation present. A sufficient number of people are necessary to can the monitor efficiently and as quickly as possible. In the case of this recent monitor, a two-shift canning operation was employed with two supervisors/shift. Sampling should occur by randomly selecting from each magazine of cigarettes approximately two handfuls and then placing them into the wire racks. When the rack is full, the cigarettes are brought to the canning station and 80 cigarettes are put into each can.

After two weeks of cold storage, cases are withdrawn as needed. Allow the cigarettes to reach room temperature prior to use (24 hours is sufficient).

E. Equipment

- electric can sealing machines (Dixie Equipment Co., Athens, Ga.)
- #2 cans with lids (80 cigt./can)
- Kimwipes (large)
- wire racks with cardboard dividers
- labels for cans
- marker pens
- pallets
- boxes (24 cans/box)

## Part II: Use of Monitor Cigarettes

The use of a Monitor cigarette has been recognized by many cigarette testing laboratories. The French norm NF V 37-003 (1975), "Comparative Determination of Tar and Nicotine in Smoke," explicitly requires that a reference cigarette be used for each smoking run:

> "The use of a Reference Cigarette is justified by the necessity to check any possible deviation in the different mechanical components of the smoking machine. This implies having a Reference Cigarette whose measured TPM value is known and stable during a given period."

Earlier references to the necessity for Monitor cigarettes are also cited in "Determination and Reporting of Total Particulate Matter, Water in Total Particulate Matter, and Nicotine in Cigarette Smoke," The Virginia Journal of Science (1967), by William Bates, Robert Griffith, et al:

> "It is common in analytical work to include check samples in order to be certain that the results obtained on unknowns are valid. This concept is especially important with smoke analytical procedures because experience, in various tobacco industry laboratories, has shown that there are unexplainable day-to-day variations in analytical results obtained on a simple sample. To obtain valid smoke analytical results on diverse samples over a period of time, it is essential to make daily checks on a carefully prepared monitor cigarette. These daily checks will indicate when variations attributable to instrumental failure or other abnormal conditions occur which would not otherwise be noted."

By strategically placing the Monitor cigarette, which has calibrated smoke deliveries, across the smoking machine ports in a cyclic rotation and statistically evaluating the corresponding results, it is possible to detect instrument problems and malfunctions which occurred during smoking. Five Monitor cigarettes are smoked per port and four ports per run analyzed together with the unknown samples. This cyclic rotation allows the Monitor to be smoked in all twenty ports over a five run cycle. The Monitor cigarettes are treated identically as the sample cigarettes throughout the testing process.

The Monitor is manufactured in an R&D environment, under tighter control than commercial brands, and is produced in comparatively smaller lots. This latter aspect allows cigarettes to be

manufactured on one making machine with selected lots of the various components (tobaccos, filters, papers, adhesives, etc.). Therefore, the inherent variability of the components and of the manufacturing process can be kept at a minimum, with a favorable impact on the Monitor Cigarettes' variability. After production, the cigarettes are equilibrated under standard conditions, sealed in cans and retained in cold storage (40°F) until used. The Monitor Cigarettes are not allowed to freeze because condensation occurs when bringing the cigarettes to ambient conditions. The preparation of a monitor cigarette that will be used on a smoking run is as follows:

1. Remove sufficient cans of Monitor cigarettes from cold storage for one week of testing (each can contains approximately 80 cigarette).

2. Allow the unopened cans to come to room temperature (apprxoximately 12 hours).

3. Open the cans and remove the cigarettes. Mark the cigarettes to the specified butt length and 9 mm insertion depth. Place the marked cigarettes into open mesh trays. Place the trays in FTC (75°F, 60% RH) and condition for at least 24 hours.

4. The Monitor cigarette must be used on each smoking run. To ensure randomization of the Monitor cigarette across the smoking machine ports, a cyclic rotation plan should be followed. The cycle rotation example shown below is designed for a 20-port smoking machine. The rotation pattern should also be followed for an 8-port smoking machine.

Acceptance limits for the Monitor smoke deliveries are established and used as acceptance/rejection criteria for the smoking runs. If the Monitor deliveries are not acceptable, all results for the corresponding smoking run must be rejected. Smoking analysis must be repeated for the brands in order to replace the rejected data with reliable ones.

Control chart analysis of the Monitor results is used to diagnose shifts in the analytical process. Monitor cigarettes allow for reproducible smoke deliveries to be determined over time. The Monitor data provide a common reference point for comparison among laboratories.

<u>Cyclic Rotation of Monitors and Brands</u>

| Port #1 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 | 16 | 17 | 18 | 19 | 20 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Run # | | | | | | | | | | | | | | | | | | | | |
| 1 | M | A | B | C | D | M | A | B | C | D | M | A | B | C | D | M | A | B | C | D |
| 2 | D | M | A | B | C | D | M | A | B | C | D | M | A | B | C | D | M | A | B | C |
| 3 | C | D | M | A | B | C | D | M | A | B | C | D | M | A | B | C | D | M | A | B |
| 4 | B | C | D | M | A | B | C | D | M | A | B | C | D | M | A | B | C | D | M | A |
| 5 | A | B | C | D | M | A | B | C | D | M | A | B | C | D | M | A | B | C | D | M |

M = Monitor Cigarette
A = Brand A
B = Brand B
C = Brand C
D = Brand D

## Part III: Statistical Procedures - Monitor Calibration

### A. Introduction

The following is a description of the statistical procedure for obtaining the values necessary for the application of statistical process control (SPC) methods in a smoking laboratory. There are two unique situations in the application of SPC in a smoking laboratory. The first is the monitor itself and the second is the manner in which the monitor is used.

The monitor is a specimen cigarette produced in large volume under controlled conditions. This leads to a very uniform product which is used to "control" the laboratory smoking process. However, there are no set reference values for the results from the smoking of a monitor. It is therefore necessary to establish a set of means and control limits for the smoking analyses each time a monitor change is made. Regardless of whether the monitor is to be used by one or several laboratories, each laboratory will need to do a study to establish the control limits for the analyses. This is necessary because equipment and conditions vary from laboratory to laboratory. For a monitor which is to be used by a group of laboratories it is best to establish the means through an interlaboratory study which will also provide some measures of the variation within and between laboratories. For a monitor which is to be used by only one laboratory the processes of establishing means and control limits may be combined in one study.

The monitor is used to determine the acceptability of results from a smoking machine run. The assumption is made that the monitor results represent the entire run, as though they were a sample of a run consisting entirely of monitor cigarettes. Although it may not be immediately obvious, this is an analogous process to selecting a random sample from a lot of items to test for acceptability of the lot. A randomization procedure is used to assign ports to the samples and monitor so that the monitor ports are interspersed among the other samples. The monitor ports then become a random sample of all the ports in the run. The analysis means and ranges from the monitor ports can then be used to determine whether the run conforms to the means and limits determined in the calibration process for the monitor. Even though there are other cigarette samples in the run, this is still analogous to taking and testing a small random sample to determine the acceptability of a large lot of items.

The following paragraphs give the procedures for calibration of a new monitor. Both single and multiple laboratory cases are covered. The procedures described are those for 20-port smoking machines with 4 ports per run allocated to the monitor. The procedures are applicable to both the FTC and ISO smoking methods.

B. Single Laboratory Calibration

Data are collected on 30 runs where the new monitor is smoked as if it were a sample with 4 ports per run. These data should be collected over a period not to exceed 2 weeks and should come from 2 or more smoking machines if possible. The time period for the collection of calibration data should be scheduled during a period when the laboratory operations will not be subject to frequent shutdowns and startups. Periods including holidays, major facility maintenance and general equipment maintenance should be avoided. The mean and range for each run should be calculated together with the grand mean and average range (r-bar). From r-bar the following preliminary 4-port estimates are calculated:

UCL mean = mean + (r-bar*0.729)

LCL mean = mean - (r-bar*0.729)

UCL r-bar = r-bar*2.282

s-hat = r-bar/2.059

The run ranges and means are then plotted and checked against these estimates. If any ranges are outside the UCL r-bar those runs are dropped and the grand mean and r-bar recalculated. New 4-port estimates are calculated and the remaining ranges checked against the new UCL r-bar. This process is repeated until all remaining ranges are within the last calculated UCL r-bar. The results are the final 4-port estimates.

If more than 10 runs have been eliminated, additional data must be collected, and the smoking and analytical processes checked for possible sources of extreme variation within a run. *Under no circumstances should less than 20 runs be used to establish the final estimates of the grand mean and r-bar.*

If an examination of the plot of ranges, r-bar and UCL r-bar shows that the value of r-bar is still inflated (r-bar is greater than two-thirds of the individual ranges) the median range should be used to calculate the final 4-port estimates. The only calculation changes are the substitution of r-median for r-bar and:

s-hat = r-median/1.978.

The s-hat values are used to determine whether a single port is a statistical outlier (see below).

It is necessary to calculate 3-port estimates for those runs where a single port is dropped because of mechanical failure or because it is a statistical outlier (see below). The same data set used for 4-port estimates may used for the 3-port estimates by using a table of random numbers to eliminate 1 port from each run. Only the runs used for the final 4-port estimates should be taken. The 3-port estimates are calculated from the following:

UCL mean = grand mean + (r-bar*1.023)

LCL Mean = grand mean - (r-bar*1.023)

UCL r-bar = r-bar*2.574

s-hat = r-bar/1.693

s-hat = r-median/1.588 (if necessary)

The process of screening and eliminating runs with excessively large ranges should be followed as used for the 4-port data. Similarly it may be found that the median range should be used to compensate for an inflated r-bar value. The same requirement for a minimum of 20 runs is mandatory.

It is not recommended that only 2 ports be used to accept or reject a run. However, the 2-port estimates may be calculated using the 4-port data with each run providing a pair of 2-port data points. The calculation equations are:

UCL mean = grand mean + (r-bar*1.880)

LCL mean = grand mean - (r-bar*1.880)

UCL r-bar = r-bar*3.268

It is not necessary to calculate s-hat for 2-port data.

C. <u>Multiple Laboratory Calibration</u>

When a monitor is to be used by several laboratories the mean values for the analyses should be obtained by an interlaboratory study. A protocol and statistical analysis for such a study is given in ASTM E691 Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Method. As the title indicates, the practice is usually used in the evaluation of a test method. However, the practice can also be used to determine a consensus value for the mean of a material with only slight

modification. Using the modified E691 practice will also provide estimates of the within and between laboratory variation for the monitor.

The basic modification to E691 is that only one sample is used. At least 5 laboratories should participate. The data should be obtained as 4 ports from each of 3 runs on each of three consecutive days.

The replicate data for the E691 statistical analysis are the run averages. The within and between laboratory variations calculated will include a day to day component. However, previous experience has shown that this is not likely to significantly inflate the estimates. If the calculated between laboratory variance is not significantly different from the within laboratory variance, the grand mean can be taken as the best estimate of the mean for routine use of the monitor. If there are significant differences among the laboratories some further investigation must be made to resolve the differences.

Once a mean value has been established, each laboratory should proceed to establish appropriate control limits for its own use. The procedure given above for single laboratory calibration should be used. Any significant deviations from the mean established in the interlaboratory study should be corrected by equipment or procedure adjustments before the data for calculating control limits are obtained. The means from the interlaboratory study are used as the center line of the control charts for means.

D. References

Annual Book of ASTM Standards, Vol. 14.02, ASTM, Philadelphia, PA, 1989.

Davies, O. L. and Goldsmith, P. L., Statistical Methods in Research and Production, Hafner Publishing Co., New York, NY, 1972.

Wheeler, D. J. and Chambers, D. S., Understanding Process Control, Statistical Process Controls Inc, Knoxville ,TN, 1986.

## Part IV: Statistial Procedures - Control Charts

### A. Introduction

The following is a description of the construction and use of control charts for smoking monitors. Separate control charts must be set up and maintained for each analysis and equipment combination. For instance, each smoking machine would have its own control charts for TPM.

### B. Construction

The control charts are constructed using graph paper or by computer if appropriate software is available. There are also some commercially available forms which may be used. Two control charts must be prepared for the 4-port data for each application. The first is the means chart. The center of the vertical scale of the graph is taken as the mean value from the calibration. The upper and lower limits (bounds) of the vertical graph scale are set so that the UCL and LCL for the mean are about two-thirds of the distance from the center to the bounds. The horizontal scale is simply the sequence of runs. Points on the horizontal scale should be set so that there is good visual separation and some room for run identification at each point. A minimum separation of 1/4" or 5 mm is suggested. A solid horizontal line is drawn at the calibration mean value and dotted or dashed horizontal lines are drawn at the control limit values. The second chart is for ranges. The lower bound of this chart is zero. The upper bound is set so that the UCL for the range is about three-quarters of the distance from zero to the upper bound. The horizontal scale should be the same as that used for the means chart. A solid horizontal line is drawn at the calibration value for r-bar and a dotted or dashed horizontal line is drawn at the UCL value. It may be possible to plot both charts on the same piece of graph paper if the vertical scales are carefully chosen.

### C. Use

Each control chart is started by plotting the runs used for calibration, in sequence. Each adjacent pair of points should be connected by a solid line. As new runs are added they are connected to the immediately preceding point. When a chart is filled a new chart is prepared which duplicates the old chart and the last 5 to 10 points of the old chart are transferred to the new chart.

1. Out of Control Points

**Means** - The means charts provide the primary tool for acceptance or rejection of data sets. Any mean which is outside the control limits is cause to reject the data set. No other action is allowable and no additional information is needed to take the action. All available data from the rejected set should be examined for possible cause(s) for the out of control condition. The point on the chart should be circled and a notation made beside the point. TPM and puff count means are used to accept or reject smoking runs. Nicotine and water means are used to accept or reject groups of samples analyzed for nicotine and water at the same time as the monitor samples. Nicotine and water means which are outside of the control limits *cannot* be used as the criteria to reject a smoking run. They may be used as supporting information when TPM and/or puff count means are out of control.

**Ranges** - The range charts are the secondary tools for acceptance or rejection of data sets. If the range for 4-port data is outside the upper control limit, the data should be examined to determine whether there is a general spread or whether a single point is out of line. A large range not attributable to a single errant point is indicative of non-homogenous data and the data set should be rejected. It is possible that a single port may be considerably different from the other three without any apparent cause for the difference. The estimated value of s-hat gives a criterion for determining whether such a port is a statistical outlier. A single port may be declared an outlier if the difference between it and the calibration mean is greater than 3*s-hat. If a single point is generating the out of control range, that point should be dropped and the 3-port mean and range calculated. The 3-port results should then be checked against the 3-port calibration for acceptance or rejection. When 3-port data are used the data are plotted on the 4-port charts with appropriate notation that they are 3-port values and that the acceptance or rejection is based on the 3-port calibration. If a port is found to be a statistical outlier without obvious failure, efforts should be made to determine the cause.

2. Lost Data

When one or two ports of the monitor are lost from a smoking run due to some observed failure, the 3-port and 2-port calibration values may be used for accepting or rejecting the data set. The 2-port data should be used only when the other two ports have been dropped for some known cause. The 2-port data should not be used when the other two ports have been deleted as statistical outliers. When only three ports are available from a run, it is allowable to

declare one a statistical outlier and use the 2-port data for acceptance or rejection. However, this should be done only when absolutely necessary and not as a matter of routine. When 2-port or 3-port data are used they should be plotted on the 4-port control charts with appropriate notations about the sample size.

3. Runs Tests

In addition to the simple in or out of control limits aspects of control charts, the control charts should be watched for unusual patterns in the data being plotted. There are two patterns which are easy to spot and important to catch. The first is a trend. A trend occurs when there is a relatively gradual but consistent change from a low level to a higher level, or the reverse. This is indicative of a "drift" in one direction of the equipment. It may also indicate a long term tendency to change in the monitor. When seven or eight sample points show an upward or downward tendency a trend is almost certainly present. There may be sufficient variation around a trend that it can only be seen when data from fifteen to twenty runs have been taken. If a trend appears in control charts for more than one piece of equipment over the same time period it is likely a monitor change. Otherwise, it is likely an equipment change. In either case an investigation should be made so that corrective action can be taken before the process goes out of control. The second pattern is a run of eight successive points all on the same side of the center line. This indicates a shift in the process mean (or range). This type of pattern is significant regardless of the apparent magnitude of the shift, it is sufficient reason to start an immediate investigation to determine the cause of the shift and apply corrective measures. If this is observed in several pieces of equipment at the same time it may be prudent to check the calibration of the monitor. However, as a general rule, an environmental change which affects the entire laboratory could cause trends or shifts similar to those caused by changes in the monitor. Whenever a monitor or general conditions change is suspected, both should be investigated.

A reproducible oscillating pattern similar to a sine-wave may be seen. The causes of such a pattern should be determined and corrected. If left alone the amplitude of the oscillations will usually increase over time and may lead to out of control values.

It should be noted that most runs tests are indicative of a coming problem. This allows the user of the control charts to be proactive and prevent out of control situations from occurring.

4. <u>Other Control Limits</u>

Two other control limit values are sometimes used on control charts. The first are commonly used on British control charts as the "inner control limits". The usual upper and lower control limits represent plus or minus three standard errors around the grand means. The inner control limits represent plus or minus two standard errors around the grand means. The following calculations are used to find the inner control limits.

4-port data -

UICL mean = mean + (r-bar*0.486)

LICL mean = mean - (r-bar*0.486)

UICL r-bar = r-bar*1.855

3-port data -

UICL mean = grand mean + (r-bar*0.682)

LICL Mean = grand mean - (r-bar*0.682)

UICL r-bar = r-bar*2.049

2-port data -

UICL mean = grand mean + (r-bar*1.254)

LICL mean = grand mean - (r-bar*1.254)

UICL r-bar = r-bar*2.512

These limits are used as early warnings of shifts in the means or ranges. If two out of three consecutive points are outside the same inner control limit the process is going out of control. The second set of limits represent one standard error around the grand means. They are calculated as follows.

4-port data -

U1σL mean = mean + (r-bar*0.243)

$L1\sigma L$ mean = mean - (r-bar*0.243)

$U1\sigma L$ r-bar = r-bar*1.427

3-port data -

$U1\sigma L$ mean = grand mean + (r-bar*0.341)

$L1\sigma L$ Mean = grand mean - (r-bar*0.341)

$U1\sigma L$ r-bar = r-bar*1.525

2-port data -

$U1\sigma L$ mean = grand mean + (r-bar*0.627)

$L1\sigma L$ mean = grand mean - (r-bar*0.627)

$U1\sigma L$ r-bar = r-bar*1.756

If four out of five consecutive points fall outside the same limit the process is going out of control. Neither the inner control limits nor the one standard error limits are usually plotted on the control charts. They are calculated and kept as reference data to detect process shifts which usually precede an out of control situation. Using all of the runs tests there is a progression of indications of an out of control process. The following is a list of rules and responses which should be initiated.

1. A single point outside the usual ($3\sigma$) control limits,
   a. Reject run,
   b. Look for cause(s) and
   c. Initiate a rerun.

2. Two out of three consecutive points outside the same inner ($2\sigma$) control limit,
   a. Investigate causes of abnormal behavior.
   b. Monitor next samples carefully for other signs of problems.

3. Four out of five consecutive points outside the same $1\sigma$ limit, and
   a. Investigate causes of abnormal behavior.
      In particular look for reason for a shift in the mean.
   b. If the next samples show similar signs of problems initiate procedures for
      compensation of the shift.

4. Eight consecutive points all on the same side of the center line.
    a. Immediately take action to compensate for the shift.
    b. Initiate an immediate investigation into causes of the shift.
    c. Consider the possibility of a need to recalibrate

Although only the first rule provides grounds for rejection, any of the other three are justification for an immediate investigation to determine the reason(s) for the apparent shift in process mean or range. As noted above, the last three rules allow the user of the control charts to be proactive by observing and correcting situations before the lack of control results in rejection of data.

# Part V: Examples

The following examples use typical data taken from monitor studies. Only the data and control charts for TPM are shown. The procedures are applicable without modification to the other smoking analyses.

*Example 1. - Single Laboratory Calibration*

Raw Data

| Run | Ports | | | | Run Mean | Run Range |
|---|---|---|---|---|---|---|
| 1 | 21.5 | 21.2 | 23.3 | 22.0 | 22.000 | 2.1 |
| 2 | 21.5 | 23.3 | 22.2 | 22.8 | 23.325 | 2.8 |
| 3 | 22.4 | 25.1 | 22.3 | 23.5 | 22.025 | 1.5 |
| 4 | 22.2 | 24.2 | 24.0 | 22.8 | 22.450 | 1.8 |
| 5 | 21.2 | 21.9 | 22.7 | 22.3 | 23.300 | 2.0 |
| 6 | 22.4 | 24.2 | 22.4 | 24.1 | 23.275 | 1.8 |
| 7 | 22.0 | 21.3 | 22.9 | 23.0 | 22.300 | 1.7 |
| 8 | 23.3 | 23.1 | 21.1 | 21.9 | 22.250 | 2.1 |
| 9 | 22.5 | 23.0 | 20.9 | 22.6 | 22.100 | 0.7 |
| 10 | 23.2 | 22.6 | 22.2 | 22.7 | 22.350 | 2.2 |
| 11 | 22.0 | 22.3 | 21.7 | 22.4 | 22.675 | 1.0 |
| 12 | 22.8 | 19.3 | 24.0 | 22.5 | 22.150 | 4.7 |
| 13 | 21.7 | 23.5 | 22.9 | 23.9 | 23.000 | 2.2 |
| 14 | 22.7 | 23.1 | 23.7 | 23.6 | 21.850 | 1.6 |
| 15 | 21.3 | 22.9 | 21.6 | 21.6 | 22.725 | 2.0 |
| 16 | 23.4 | 22.8 | 22.4 | 21.9 | 23.275 | 1.0 |
| 17 | 21.6 | 22.6 | 23.1 | 23.6 | 22.625 | 1.5 |
| 18 | 24.5 | 22.6 | 21.9 | 23.4 | 23.100 | 2.6 |
| 19 | 20.9 | 22.6 | 22.1 | 22.9 | 22.125 | 2.0 |
| 20 | 23.8 | 18.2 | 22.0 | 22.5 | 22.850 | 1.9 |
| 21 | 21.8 | 23.7 | 22.3 | 23.6 | 22.575 | 0.4 |
| 22 | 23.0 | 21.7 | 23.4 | 20.0 | 21.625 | 5.6 |
| 23 | 22.4 | 22.4 | 22.8 | 22.7 | 22.025 | 3.4 |
| 24 | 23.0 | 21.0 | 21.6 | 22.4 | 22.000 | 2.0 |
| Grand Mean | | | | | 22.499 | 2.1 |

Trial limits

UCL mean = 22.499 + (2.1*0.729)
= 22.499 + 1.531 = 24.030

LCL mean = 22.499 - (2.1*0.729)

= 22.499 - 1.531 = 20.968

UCL range = 2.1*2.282 = 4.792

Run No. 22 is out of limits on range and is dropped.

Grand Mean (23 runs) 22.537 1.948

Second trail limits

UCL mean = 22.537 + (1.948*0.729)
= 23.957

LCL mean = 22.537 - (1.948*0.729)
= 21.117

UCL range = 1.948*2.282 = 4.445

Run No. 12 is out of limits on range and is dropped.

Grand Mean (22 runs) 22.555 1.832

Third trial limits

UCL Mean = 22.555 + (1.832*.729)
= 23.896 = 23.9

LCL mean = 22.555 - (1.832*0.729)
= 21.219 = 21.2

UCL range = 1.832*2.282 = 4.1681 = 4.2

s-hat = 1.832/2.059 = 0.9

Since all ranges and means are now within the trial limits, and the trial limits are based on more than 20 runs, the means and trial limits are accepted as the initial calibration values.

The parameters for 3-port and 2-port run sizes are calculated in the same way by using subsets of the raw data. If the port assignments are made in pairs (*e.g.* 3,7,13,17),there is a natural pair of 2-port rational subsets ( *e.g.* 3,13 and 7,17). For the 3-port sets a table of random numbers or a computer random sampling routine may be used to select 3 of the 4 ports from each run. Runs

already excluded from the 4-port calibration should be excluded before selection of the 3-port and 2-port subsets. The 3-port and 2-port subset should be independently selected. Since runs may have already been excluded before selection of the subsets care must be taken not to use less than 20 run in the calculation. In the above example there are 22 runs available for the selection of the 2-port and 3-port subsets. In either case if more than 2 runs should be dropped additional runs would have to be obtained to complete the calibration.

***Example 2 : Multiple Laboratory Calibration***

The following example is taken from data from 5 laboratories. Each laboratory submitted raw data from 9 runs made on 2 machines over a 3 day period. The 4-port averages were used as individual replicates from each laboratory.

Interlaboratory Test Results
(ASTM E691 Analysis)

Laboratory TPM Means

| Lab 1 | Lab 2 | Lab 3 | Lab 4 | Lab 5 |
|---|---|---|---|---|
| 22.708 | 22.558 | 23.192 | 22.500 | 23.433 |

Laboratory Standard Deviations

| Lab 1 | Lab 2 | Lab 3 | Lab 4 | Lab 5 |
|---|---|---|---|---|
| 0.608 | 0.574 | 0.354 | 0.345 | 0.663 |

Laboratory Deviations from Sample Means

| Lab 1 | Lab 2 | Lab 3 | Lab 4 | Lab 5 |
|---|---|---|---|---|
| -0.170 | -0.320 | 0.313 | -0.378 | 0.555 |

Between Laboratory Consistency Statistics (h)

| Lab 1 | Lab 2 | Lab 3 | Lab 4 | Lab 5 |
|---|---|---|---|---|
| -0.41 | -0.78 | 0.76 | -0.92 | 1.35 |

* number exceeds 5% critical value ( 1.74)
? number is greater than 85% of critical value

Within Laboratory Consistency Statistics (k)

| Lab 1 | Lab 2 | Lab 3 | Lab 4 | Lab 5 |
|---|---|---|---|---|
| 1.16 | 1.09 | 0.67 | 0.66 | 1.26 |

* number exceeds 5% critical value ( 1.53)
? number is greater than 85% of critical value

Sample Summary

| Mean | S(x-bar) | Sr | SR | r | R |
|---|---|---|---|---|---|
| 22.878 | 0.412 | 0.526 | 0.645 | 1.473 | 1.806 |

The results show that:

a. The grand mean was 22.9 mg TPM.

b. All laboratories were acceptable for within and between laboratory consistency (no "k" or "h" statistic even approached significance at the 5% level).

c. The pooled within laboratory standard deviation (Sr) is 0.526.

d. The difference between two runs in the same laboratory should be greater than 1.5 (r) only about 5% of the time. Note that this is a between run measure and cannot be used in place of the average range or directly compared to the average range.

e. The pooled between laboratory standard deviation (SR) is 0.645.

f. The difference between two runs from two different laboratories should be greater than 1.8 (R) only about 5% of the time.

Using the Sr value of 0.526 an estimate of the pooled average range for 4 ports can be calculated as:

Est. r-bar = 2*2.059*Sr = 2.2

This estimate is likely to be differ from that observed for any individual laboratory because it is calculated from a pooled value. The data are also taken from a much smaller number of runs than should be used for control chart calibration. In addition, this estimate may be high because the analysis uses all data and there has been no screening of individual run range data to eliminate those runs which could create an inflation of the average range.

Since the next step would be for each laboratory to do a calibration, comparisons such as these can be used obtain a preliminary estimate of the individual laboratory results. The data used for Laboratory 2 in this analysis is a subset of the data used in Example 1. Even though the analyses are quite different, and no special selection of data was made, the results are reasonably consistent for Laboratory 2: mean 22.6 *vs* 22.6, between run S.D. 0.45 *vs* 0.57.